

Final Report to AFOSR
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by
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Objectives :

The objective of this program is as follows :

- *Process solutions to achieve high critical current in thick-film HTS 2G wires, and*
- *Development of conductor with improved magnetic field performance.*

Status of Effort :

This was a short (less than 1 year) program to build on the success of the previously funded AFOSR Contract FA9550-04-C-0020. In the last year of this program, we made major progress in both objectives of the program. We fabricated films of different thickness by our standard MOCVD processes and worked with our collaborators (ORNL, LANL, FSU,) to understand the microstructural reasons for the I_c performance using a variety of advanced characterization tools. We then modified our MOCVD process using a multipass technique to improve I_c performance in thick films. We modified rare-earth composition to improve critical current performance.

Experimental :

MOCVD of YBCO was conducted in a custom-built facility at SuperPower described in previous Progress reports. The surface morphology of the YBCO films was examined by Field Emission Scanning Electron Microscope (FESEM) followed by compositional analysis by Energy Dispersive X-ray Spectroscopy (EDS). In addition we also analyze bulk composition by Inductively Coupled Plasma (ICP) spectroscopy and elemental depth profiling by Glow Discharge Optical Emission Spectroscopy. Film cross sections were made with Focussed Ion Beam Milling (FIB). The texture of the films was analyzed by XRD including polefigure measurements. The thickness of the films was measured by surface profilometry.

Accomplishments/New Findings :

In our Final Report for Contract FA9550-04-C-0020 that ended in May 2007, we reported improvements in thickness influence on I_c of MOCVD-based 2G wires. The thickness was varied by a multipass approach where each pass was a distinct MOCVD

process run, and the subsequent layer was deposited atop the film processed in the previous pass. By this approach, we could modify the process conditions as needed in each pass. The thickness of film deposited in each pass was approximately 0.7 micron. We reported achievement of 720 A/cm over a 3.5 micron MOCVD film. We also showed microstructural features such as a-axis grain growth and compositional variation that limited the J_c of 2.8 micron thick films. It was clear from that work that microstructural complications increase with thickness. So, we focused our effort on achieving higher J_c in thinner films. In this effort, we modified the precursor chemistry from Y-Sm-based to Gd-Y-based materials.

Using Gd-Y precursors, we were able to consistently achieve J_c over 4.5 MA/cm^2 at self field in 0.7 micron thick films compared to about 4 MA/cm^2 in films of same thickness with Y-Sm precursors. Figure 1 shows a cross sectional TEM image of a 0.7 micron thick GdYBCO film. An abundance of defects can be seen both along the horizontal and vertical directions. These defects were found to be (Gd,Y) oxide similar to the (Y,Sm) oxide defects seen in films before.

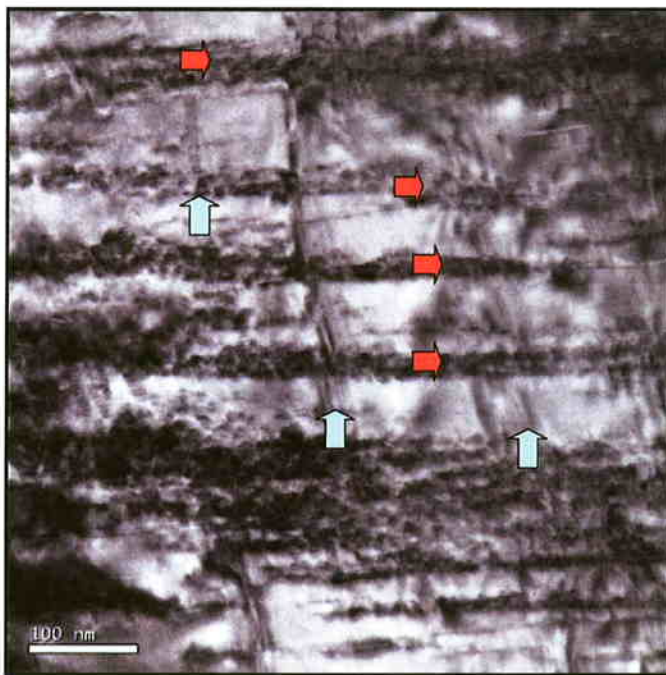


Figure 1. Cross sectional TEM image of a 0.7 micron MOCVD film made with Gd-Y-based precursors.

Figure 2 shows I_c and J_c of films of increasing thickness using the Gd-Y precursor chemistry. Results from 2007 and 2006 using Y-Sm precursor chemistry are also included for comparison. From the figure, it can be seen that higher currents can be achieved with Gd-Y precursor chemistry. In particular, a 2.8 micron thick film was grown with a critical current of 740 A/cm. This value is even better than that achieved in a 3.5 micron thick film using Y-Sm precursor chemistry. This high I_c value was measured over the entire width of tape of 12 mm without patterning using continuous dc currents. A I-V curve obtained from this measurement is shown in Figure 3. The J_c of the 2.8 micron film is 2.65 MA/cm².

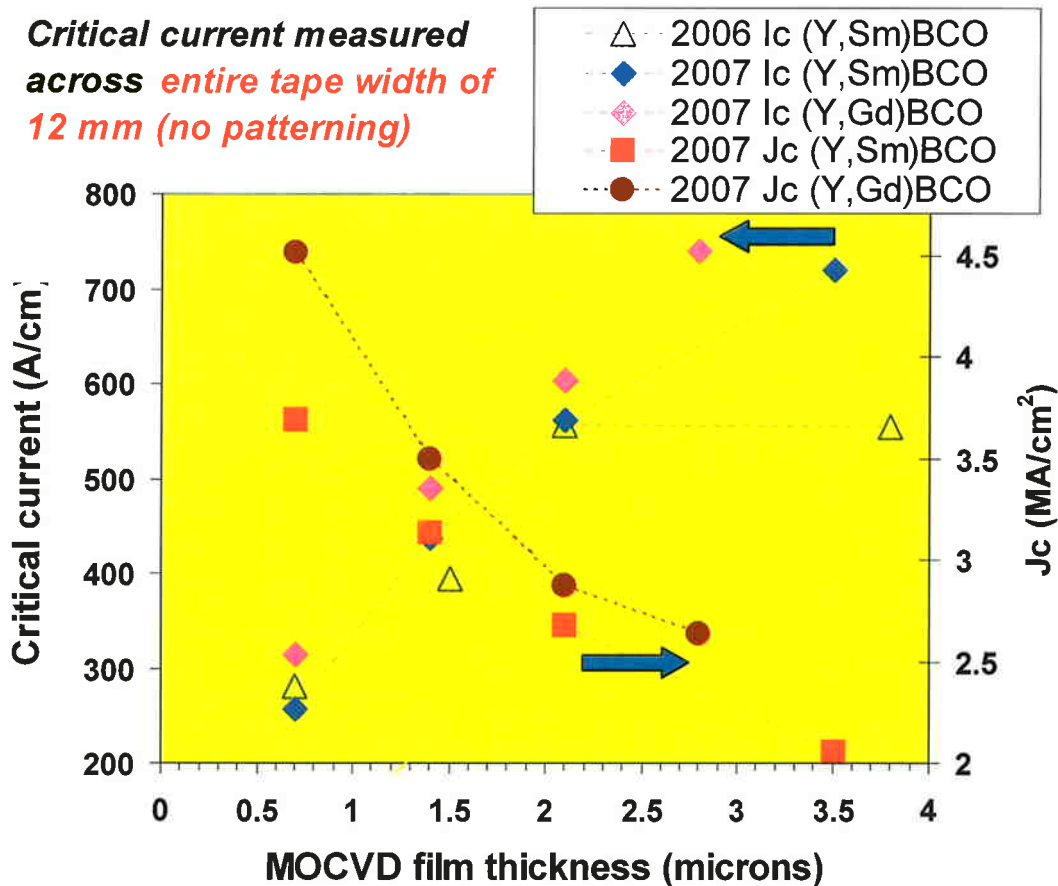


Figure 2. Critical currents and critical current densities of 2G wire fabricated by MOCVD with different YBCO layer thickness using (Gd, Y) and (Sm,Y) precursors.

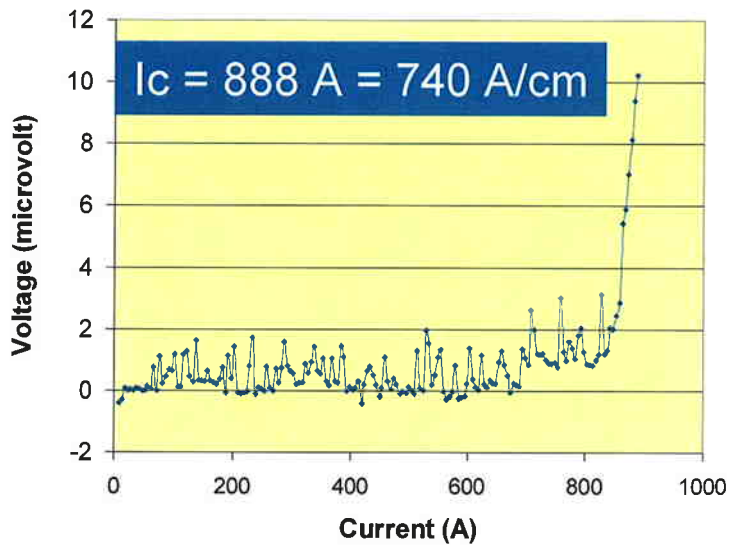


Figure 3. I - V curve obtained over a short sample of GdYBCO with 2.8 micron thick film. The measurement was conducted over the entire width of 12 mm without patterning.

Next, the in-field performance of the 2.8 micron thick GdYBCO film was examined at 77 K and 65 K and the results are shown in Figure 4 and summarized in Table I.

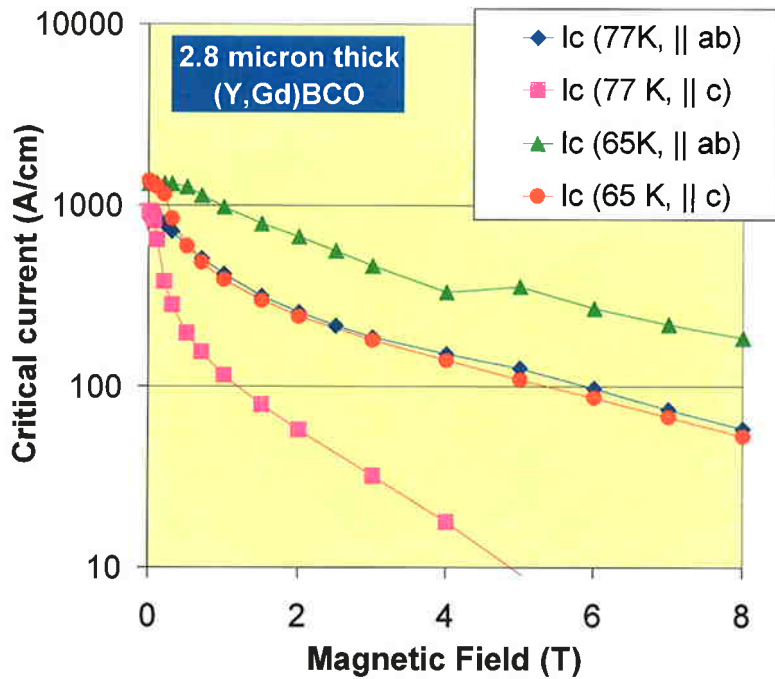


Figure 4. Critical currents over a range of magnetic fields at 77 K and 65 K of a 2.8 micron thick GdYBCO film.

Table I. Critical current and J_c values obtained at 77 K, 1 T and 65 K, 3 T of a 2.8 micron thick GdYBCO film

	I_c (A/cm)	J_c (MA/cm ²)
77 K, 1 T, B a-b	419	1.5
77 K, 1 T, B c	116	0.42
65 K, 3 T, B a-b	468	1.67
65 K, 3 T, B c	181	0.65

As shown in Table I, a high J_c is achieved especially in the orientation of field parallel to the a-b plane. An examination of the microstructure of the film (Figure 5) shows an abundance of defects both along the a-b plane and c-axis as seen in the 0.7 micron thick film (Figure 1).

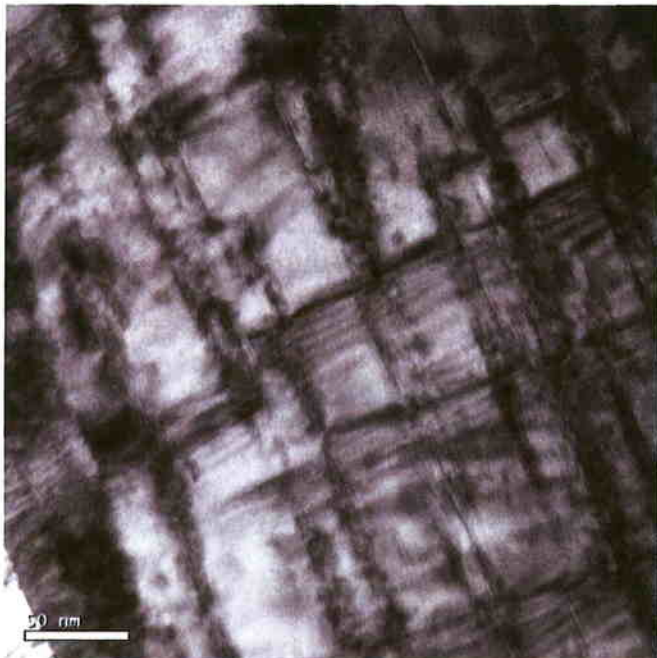


Figure 5. Cross sectional TEM image of a 2.8 micron MOCVD film made with Gd-Y-based precursors

Compositional mapping was conducted over the cross section of the 2.8 micron film and the results are shown in Figure 6. As shown in the figure, the defects along the horizontal direction were confirmed to be Gd-Y rich.

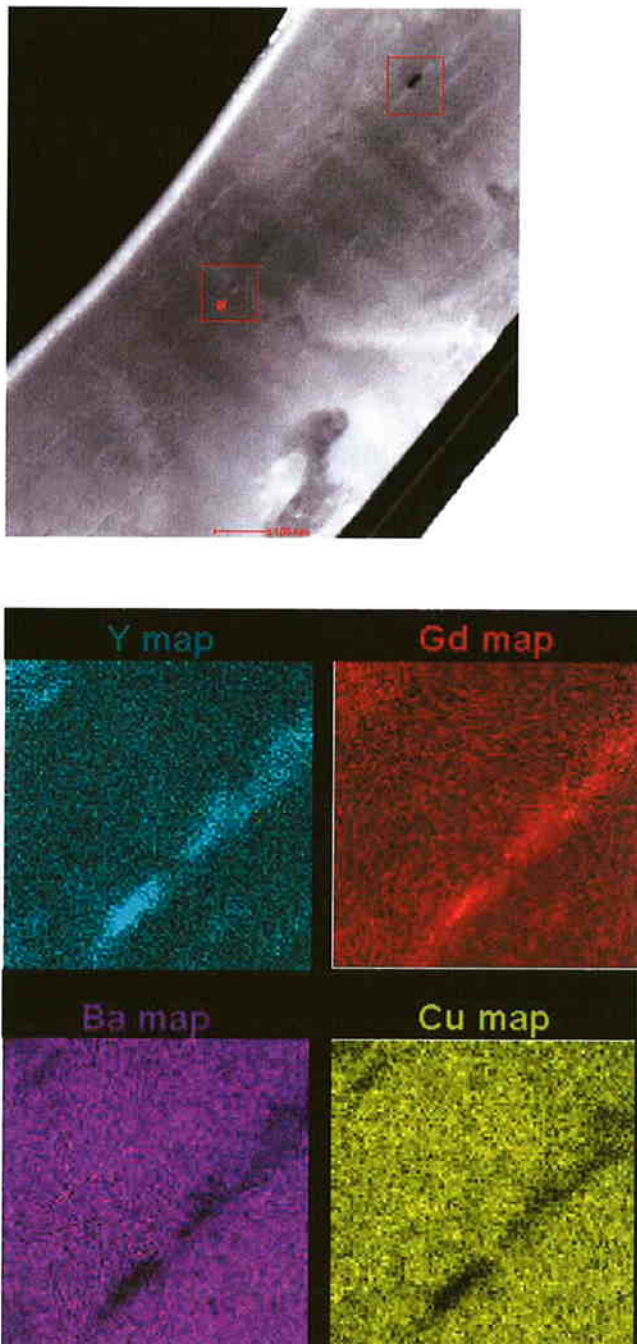


Figure 6. Compositional mapping of a 2.8 micron thick GdYBCO film.

We next explored films thicker than 2.8 microns. Additional passes each resulting in 0.7 micron thicker layers were done up to a total thickness of 5 microns. Unfortunately, not only the critical current density reduced but we found a sharp decrease in critical current itself in films thicker than 2.8 microns as shown in Figure 7. T_c measurements showed only a slight drop in T_c in thicker films (Figure 8).

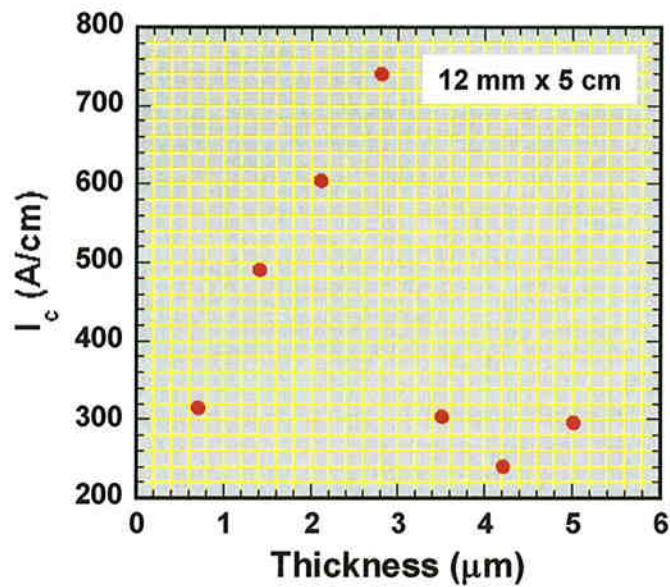


Figure 7. Critical current levels of GdYBCO films at 77 K up to 5 microns in thickness.

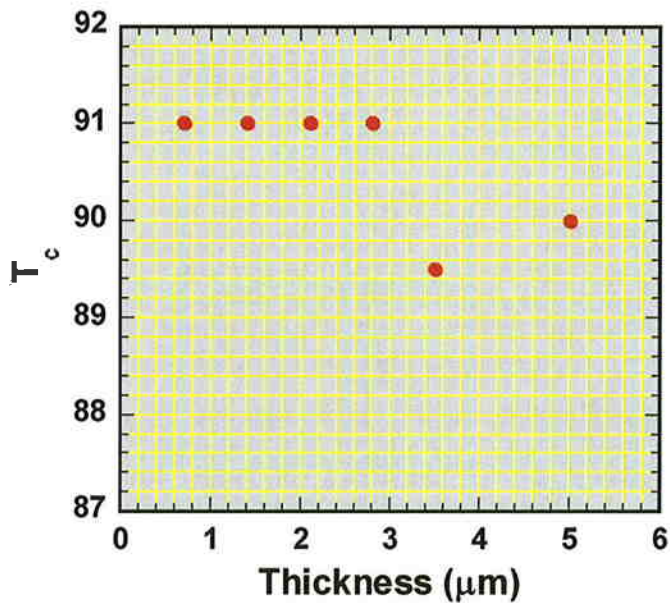


Figure 8. T_c of GdYBCO films up to 5 microns in thickness.

In order to eliminate any effect of T_c change in thicker films, critical current measurements were performed at 5 K and the results are shown in Figure 9. As shown in the figure, even at temperatures far from T_c , films thicker than 2.8 microns show lower I_c . Next, in order to understand the microstructural causes for the I_c decrease in films thicker than 2.8 microns, we studied the fraction of a-axis and 45 degree rotated grains in all films. Results from these measurements are shown in Figures 10 and 11. As shown in the figures, the a-axis fraction did not increase in films thicker than 2.8 microns. However, more 45 degree rotated grains were found in such thicker films. Also, electron diffraction results showed more polycrystalline content (or random orientation) in films thicker than 2.8 microns. Our future work in this area would be focused on reducing such misoriented grains so as to achieve higher currents in films thicker than 3 microns.

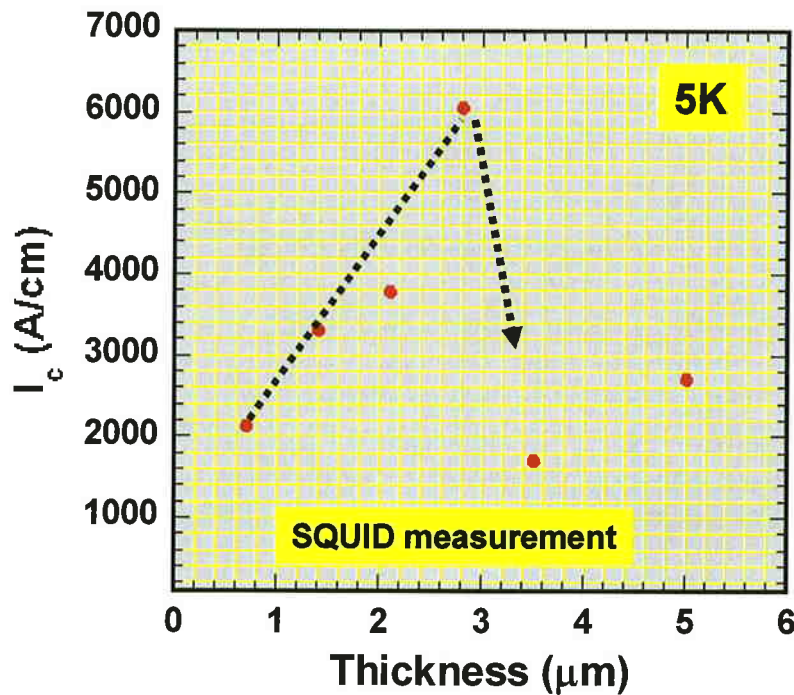


Figure 9. Critical current levels of GdYBCO films at 5 K up to 5 microns in thickness.

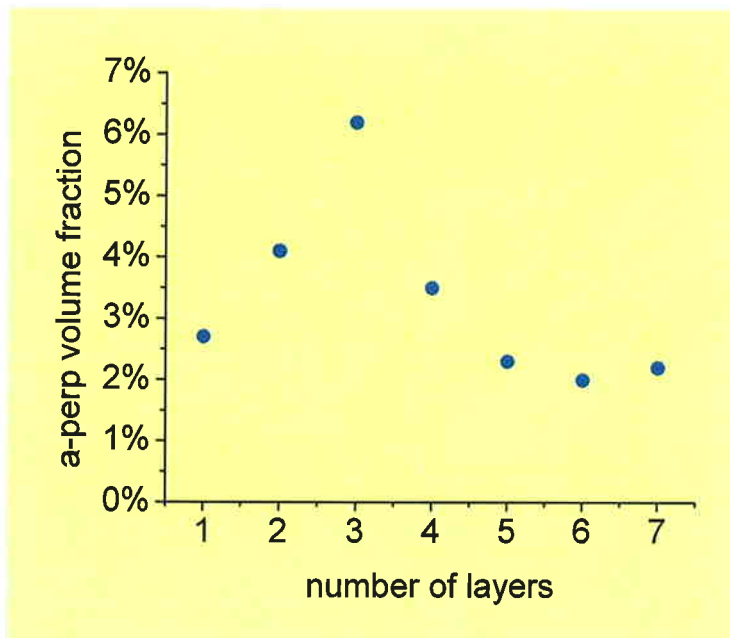


Figure 10. Fraction of a-axis grains in GdYBCO films up to 3.5 microns in thickness.

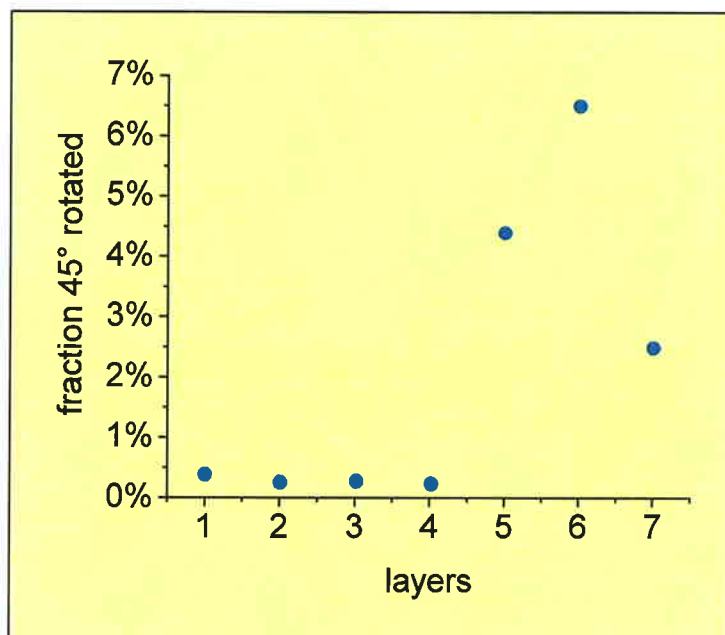


Figure 10. Fraction of 45-degree rotated grains in GdYBCO films up to 3.5 microns in thickness.

Personnel Supported :

Dr. Yimin Chen, Sr. Materials Scientist, MOCVD

Dr. Andrei Rar, Sr. Materials Scientist, Characterization

Publications :

1. V. Selvamanickam, Y. Chen, X. Xiong, Y. Xie, X. Zhanga, A. Rar, M. Martchevskii, R. Schmidt, K. Lenseth, J. Herrin, “Progress in Second-generation HTS Wire Development and Manufacturing” *Physica C*. (to be published)
2. Z. Chen, D. M. Feldmann, X. Song, S. I. Kim, A. Gurevich, J. L. Reeves, Y. Y. Xie, V. Selvamanickam and D. C. Larbalestier, “Three-dimensional vortex pinning by nano-precipitates in a Sm-doped $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ coated conductor”, *Supercond. Sci. Technol.* **20**. S205-S210 (2007)
3. A. Bayou, G. Majkic, Y.X.Zhou, V.Selvamanickam, Y.Y.Xie and K. Salama, “Effect of transverse pull loading on electromechanical properties of 2nd generation HTS wires ”, *Proc. MEM 2007, Princeton, August 2007* (submitted)
4. G. Majkic, A. Bayou , Y.X.Zhou, V.Selvamanickam, Y.Y.Xie and K. Salama, “Electromechanical properties of 2nd Generation HTS wires subjected to combined torsional and tensile loading”, *Proc. MEM 2007, Princeton, August 2007* (submitted)

Interactions/Transitions :**a. Conference Presentations :**

1. ICMC, Chattanooga, July 2007
2. DOE Peer Review, Washington D.C., August 2007
3. ISS, Tsukuba, November 2007
4. CCA, Jeju Island, November 2007

b. Interaction :

SuperPower has had extensive collaboration with ORNL, LANL, and FSU for understanding the in-field performance and to elucidate the microstructural reasons for the performance of our MOCVD-based conductors. Additional microstructural work, Raman Spectroscopy, and MOI was done together with ANL. Samples of MOCVD tapes have been provided to AFRL for various measurements. We have collaborated with Florida State University and University of Houston for stability, and mechanical property

measurements. We have worked with Ohio State University on ac loss measurements and with California State University for VTLSM. SEM, TEM, AFM, and FIB analysis have been conducted by SuperPower staff at U. Albany.

c. Transition :

The AFOSR program has had a large impact on ongoing materials and device development programs at SuperPower. *The AFOSR program has been a critical program at SuperPower for the development of YBCO 2G wire.* The success of the program has led to a high-performance and potentially lower cost replacement for Bi-2223 conductor. Bi-2223 conductor is currently the main HTS conductor available in long lengths and is used in all demonstration projects. Based on its superior performance and potential lower cost, YBCO is the clear choice for HTS conductor for all these devices. *SuperPower recognized this fact and has strongly supported the AFOSR program through funds for capital equipment including MOCVD facilities.* Last year, SuperPower committed substantial funds towards capital equipment for MOCVD facilities that include upgrades to Pilot MOCVD facility and Prototype MOCVD facility, as well as purchase of a new Pilot MOCVD facility. The AFOSR program will eventually enable the fabrication of a high performance superconducting tape that can find wide use in military, electric power, magnetic, medical and applications.

8. Inventions :

None.

9. Honors & Awards :

None